

**TITLE OF INVENTION**  
**MICRONIZATION PROCESS AND POLYMER PARTICLES PRODUCED THEREFROM**

**CROSS-REFERENCE TO RELATED APPLICATION**

This application claims priority under 35 U.S.C. §119 from U.S. Provisional Application Ser. Nos. 60/266,810 (filed February 06, 2001) which is incorporated by reference herein as if fully set forth.

**BACKGROUND OF THE INVENTION**

Field of the Invention

This invention generally relates to a micronization process of producing polymer particles suitable for use in powder coating applications, and more particularly relates to producing an aqueous slurry or powder of substantially round polymer particles.

Description of Related Art

It is known to use powder coatings containing polymer particles for coating various substrates, such as metal, wood, cement, leather, ceramic and resinous substrates. Some of the typical methods utilize an electrostatic spray application or a fluidized bed of polymer particle powder in which the substrate to be coated is fully or partially sprayed or dipped to cover the substrate surface with polymer particles. The covered surface is generally then heated to melt the polymer particles to form a coating on the substrate surface. Powder particles from thermoplastic or thermosetting polymers are typically employed. The thermosetting polymer particles can be blended with a crosslinking agent during its preparation.

Another typically employed application method includes applying aqueous slurry of polymer particles over substrate surfaces by conventional application means, such as spraying, dip coating, brush coating or roller coating. Slurry coated surfaces are dried to remove moisture and then heated to melt the polymer particles to form a coating on the substrate surface.

Several methods are employed in producing polymer powders or powder slurries. One method involves mixing polymers with other additives, melt extruding the mixture and then cryogenically grinding the extrudate. Sometimes, the ground particles are reheated in water to blunt the sharp edges present on the cryogenically ground particles. FIG. 1 illustrates the polymer particles produced by such a method. One of the problems

associated with cryogenically ground particles is they tend to have diverse particle size distribution.

Yet another method suited for producing polymer particles from a thermosetting polymer blended with a crosslinking agent is described by Kalck et al. in PCT/EP97/07109, published on June 25, 1998. The Kalck method is directed to mixing a thermosetting polymer with a crosslinking agent and then melting the mixture into a viscous liquid melt, shearing the melt in a shearing device simultaneously supplied with water and emulsifier, and then rapidly cooling the sheared particles in less than 5 seconds, preferably in less than a second. Alternatively, the molten streams of thermosetting polymer and crosslinking agent are mixed in a molten state and then the mixed viscous melt is sheared in a multi-stage mixing/shearing device simultaneously supplied with water and emulsifier. One of the problems with such a device is the viscous melt in the conveying conduits, such as pipes, can clog up the shearing device and the conveying conduits. Moreover, the conveying conduits, which have to be kept heated to keep the ingredients in a molten state also tend to impart a varying process temperature history to the polymer particles, a problem further exacerbated by the presence of stagnant spots that are invariably present in any piping system. As a result, polymer particles containing a thermosetting polymer and crosslinking agent exposed to a varying process temperature history can result in particles having varying degrees of premature crosslinking. Thus, a coating resulting from the use of a powder coating composition or powder slurry containing such polymer particles would not have uniform coating properties, such as adhesion, gloss, flow-leveling and coating life. Therefore, a need exists for a process and a clog free device that produces substantially rounded polymer particles at high through put rates but does not impart varying process temperature history. The process and the device of the present invention fully address these needs.

#### **BRIEF SUMMARY OF THE INVENTION**

The present invention is directed to a process for producing substantially round polymer particles comprising the steps of:

- (i) forming a mixture comprising polymer pellets, an aqueous medium and at least one surfactant;
- (ii) heating said mixture under plug flow and plug free heating conditions;
- (iii) shearing heated mixture; and

(iv) cooling sheared mixture under plug free cooling conditions to produce said substantially round polymer particles.

The present invention is also directed to a device for producing substantially  
5 round polymer particles comprising:

(i) means for forming a mixture of polymer pellets, an aqueous medium and at least one surfactant;

(ii) means for heating said mixture under plug flow and plug free heating conditions;

10 (iii) means for shearing said heated mixture; and

(iv) means for cooling said sheared mixture under plug free cooling conditions.

One of the advantages of the process and device of the present invention is to produce thermosetting polymer particles that can comprise a thermosetting polymer,  
15 crosslinking agent, and surfactant and, if desired other suitable ingredients wherein the particles have substantially identical and uniform process temperature history. As a result, the degree of premature crosslinking does not substantially vary from one particle to another. Thus, coatings resulting from the use of such particles have predictable and uniform coating properties, such as adhesion, crack resistance, coating life, flow-leveling,  
20 etch and mar resistance. Plug flow conditions utilized in the present invention during heating and preferably during cooling of polymer ensures the aforescribed advantage.

The plug flow conditions used during the heating of the polymer pellets also ensure uniform shearing of the heated polymer pellets, since the pellets are at uniform process temperature under the plug flow conditions. As a result, the shearing step  
25 becomes more predictable and uniform resulting in polymer particles having more rounded shapes as compared to conventional systems.

Another advantage of the process and device of the present invention is to produce polymer particles having desired particle size and particle size distribution in the form of either an aqueous particle slurry or a powder, either of which are especially suitable for  
30 use in the OEM automotive coatings and refinishes, and in industrial coatings.

Still another advantage of the process and device of the present invention over conventional systems is to produce thermosetting or thermoplastic polymer particles at

high rates of productions with substantially no clogging either in polymer conveying systems or in shearing devices.

Yet another advantage of the process and device of the present invention over conventional systems is the ease in producing either thermoplastic or thermosetting polymer particles with substantially no downtime required in switching from one type of product to another. The process and device of the present invention also advantageously allows one to rapidly switch with substantially no downtime from one pigmented polymer particle to another color or to rapidly switch from one type of polymer to another.

### **BRIEF DESCRIPTION OF THE DRAWINGS**

FIG. 1 is a magnified view of the polymer particles produced by one of the conventional processes.

FIG. 1A is a schematic diagram of a device of the present invention used in the process of the present invention.

FIG. 2A is a plan view of a heating zone used in means for heating of the device.

FIG. 2B is a cross-sectional elevation view of the means for heating taken along the cross section 2B-2B in FIG. 2A.

FIG. 2C is a partial cross-sectional elevation view of the means for heating taken along the cross section 2C-2C in FIG. 2A.

FIG. 2D is a partial end view of a flange cover of a heating zone of the means for heating.

FIG. 2E is a partial three-dimensional view of the flange cover to illustrate the spatial positioning injection ports of the heating zone.

FIG. 3A is an alternate embodiment of the means for cooling used in the device of present invention.

FIG. 3B is a magnified end view of the means for cooling of FIG. 3A.

FIG. 3C is a partial cross-sectional elevation view of the means for cooling of FIG. 3B taken along the cross section 3C-3C in FIG. 3B.

FIG. 4 is a magnified view of the polymer particles of the present invention produced in Example A.

FIG. 5 is a magnified view of the polymer particles of the present invention produced in Example B.

FIG. 6 is a magnified view of the polymer particles of the present invention produced in Example C.

FIG. 7 is a magnified view of the polymer particles of the present invention produced in Example D.

FIG. 8A is a magnified view of the polymer pellets used for producing the polymer particles in Example E.

FIG. 8B is a magnified view of the polymer particles of the present invention produced in Example E.

FIG. 9A is a magnified view of the polymer pellets used for producing the polymer particles in Example F.

FIG. 9B is a magnified view of the polymer particles of the present invention produced in Example F.

FIG. 10A is a magnified view of the polymer pellets used for producing the polymer particles in Example G.

FIG. 10B is a magnified view of the polymer particles of the present invention produced in Example G.

**DETAILED DESCRIPTION OF THE INVENTION**

As used herein:  
“Plug free condition” means a flow condition through a pipe of polymer pellets in an aqueous medium wherein the pellets pass through a pipe having a substantially smooth bore and having substantially no obstructions or projections therein.

“Plug flow condition” means a flow condition through a pipe of polymer pellets in an aqueous medium wherein each polymer pellet passing through the pipe spends the same length of time in the pipe. As a result, the residence time history of the pellets during a heating step is substantially identical, thus substantially minimizing or eliminating variation in the degree of premature crosslinking of one thermosetting polymer pellet to the adjacent polymer pellet passing though the pipe. Similarly when a plug flow cooling condition is opted in cooling the aqueous medium containing polymer particles produced after shearing, the residence time history of the polymer particles during the cooling phase would be substantially identical. Thus, one can substantially minimize or eliminate variation in the degree of premature crosslinking of one

thermosetting polymer particle to the adjacent polymer particle passing through the pipe during cooling.

One of the conditions for achieving a plug flow during the heating or cooling steps is to ensure that the flow is turbulent and not laminar. Turbulence of a flow is ascertained by determining its Reynolds Number (See generally V. L. Streeter and E. B. Wylie, Fluid Mechanics, 7<sup>th</sup> edition, McGraw-Hill Book Company, New York, 1979, pp. 195-198). The Reynolds Number of a fluid flowing through a pipe is calculated by using the following equation:

$$R = \frac{VD}{\mu}$$

where R is the Reynolds Number, V is the transportation velocity, D is the inner diameter of the pipe and  $\mu$  is the kinematic viscosity of the fluid passing through the pipe. A flow changes from laminar flow to a turbulent flow when Reynolds numbers range from 2000 to 4000. Reynolds numbers suitable for use in the present invention vary in the range of 2,000 to 500,000, preferably in the range of from 5,000 to 200,000.

Another critical requirement for the plug flow to occur is that a minimum transportation velocity must be achieved. This requirement is set by the Durand's minimum transportation velocity:

$$V_M = F \left[ 2gD \left( \frac{\Delta\rho}{\rho_{medium}} \right) \right]^{0.5}$$

where  $V_M$  is the minimum transportation velocity, F is an empirically derived constant that depends on the size of a polymer particle being conveyed, g is the acceleration due to gravity, D is the inner diameter of the pipe,  $\Delta\rho$  is the absolute value of the difference between the densities of the polymer pellets and the aqueous medium and  $\rho_{medium}$  is the density of the aqueous medium. (See generally J. T. Davis, Calculation of Critical Velocities to Maintain Solids in Suspension in Horizontal Pipes, Chemical Engineering Science, v. 42, no. 7, 1987, 1667-1670). Typical values suitable for use in the present invention for F are in the range between 0.9 and 1.4, preferably in the range of 0.95 to 1.3; the coarser the particles the higher the F number and the higher will be the transportation velocity. Thus, by determining the Reynolds Number and Durand's

minimum transportation velocity, an appropriate inner diameter of the pipe for a desired rate of production can be sized to produce plug flow.

“Amorphous polymers” are polymer molecules that are intertwined and arranged randomly. Most thermosetting polymers are amorphous.

“Crystalline polymers” are polymer molecules that are arranged closely and in a discernible order.

“Semi-crystalline polymers” are polymer molecules that include crystalline regions within an amorphous matrix. Most thermoplastic polymers are crystalline or semi-crystalline polymers.

Below a temperature known as the glass transition temperature ( $T_g$ ), the molecules of a polymer material are frozen into a glassy state and above  $T_g$  the amorphous polymers enter a rubbery state in which the material becomes plastic or even liquid. In the case of crystalline polymers the liquid state is not reached until the melting temperature ( $T_m$ ) is passed.

“Roundness” of a polymer particle is related to the overall shape of a polymer particle. Substantially round polymer particles produced by the process of the present invention have no sharp edges. As a result, substantially round polymer particles when charged electrostatically stick more evenly to a surface being electrostatically coated with polymer particles than the particles having sharp edges. Preferably, a substantially round particle has a substantially spherical shape.

“Particle size distribution” relates to the population density of a particle of a desired size in the population of polymer particles under study. Thus, a number close to 100 indicates that all of the particles would have the same size. Therefore, the higher the particle size distribution number, the more uniform will be the particle size. In the present invention, the particle size distribution varies from 1 to 100, preferably from 10 to 100 and more preferably from 20 to 100.

It should be noted that for the sake of clarity the same components are referred to by the same Arabic numerals throughout all the Figures referred to herein.

The schematic diagram in FIG. 1A illustrates the various components used in a device 1 in the present invention.

In its broadest aspects, device 1 for producing substantially round polymer particles includes:

(i) means 3 for forming a mixture of polymer pellets, an aqueous medium and at least one surfactant;

(ii) means 5 for heating the mixture under plug flow and plug free heating conditions;

(iii) means 7 for shearing the heated mixture; and

(iv) means 9 for cooling the sheared mixture under plug free cooling conditions.

Means 3 for forming include a slurry tank 10 into which polymer pellets, preferably stored in a hopper 12, are conveyed through a pellet intake 14. If desired, the delivery of the polymer pellets from hopper 12 can be automated and regulated by conveying the pellets via a conventional screw feeder (not shown) positioned between hopper 12 and pellet intake 14. Intakes 16 and 18 through which water, preferably deionized, and surfactant are respectively conveyed to slurry tank 10. If desired, tank 10 can be provided with additional intakes for conveying other suitable components, such as pigments, viscosity modifier, and water miscible co-solvents, to the aqueous medium in tank 10. Preferably, chemically inert materials, such as fiberglass or polyethylene, are used in the fabrication of tank 10. The contents in tank 10 are preferably agitated with a conventional stirrer 20, such as an air driven four-bladed impeller.

It should be understood that the order in which the mixture is formed in slurry tank 10 is not critical. For example, it is contemplated that the polymer pellets can include one or more surfactants blended therein, which are then conveyed to slurry tank 10. Alternatively, one can also add the same or yet another surfactant to slurry tank 10 along with the aforesaid polymer pellets that include the surfactant blended therein.

A suction port of metering pump means 22 is connected to tank 10 to convey the mixture, under pressure, from tank 10 to heating means 5 via delivery port 24. Preferably, metering pump means 22 include a positive displacement pump having a rotor in a screw design configuration, all covered with nitrile rubber. One suitable pump available from Philip H. Kline & Co., Inc.; Pottstown, Pennsylvania is Moyno® pump Model 6M2 with # 3 drive end. If desired, metering pumping means 22 may include more than one pump connected in series to increase the pumping pressure in stages. Means to measure the flow rate and the pressure (not shown) of the mixture supplied by metering pump means 22 is monitored to attain the plug flow condition.



Means 5 for heating include a plurality of heating sections 26 interposed with a plurality of heating zones 28. Preferably, for the sake of uniformity and flexibility, substantially identical plurality of heating sections 26 and heating zone 28 are utilized.

5 However, if desired heating sections 26 and heating zones 28 of unequal length or sizes can be utilized. Heating section 26 has a substantially elongated shape, preferably tubular, to permit the aqueous medium to flow therethrough without substantially any obstruction, i.e., under a plug free condition. Pipes having substantially no obstruction and smooth bore are preferred. As a result, polymer pellets in the mixture can be heated  
10 at substantially high throughput rates. Heating section 26 is preferably a transparent tube that permits the user to make a visual inspection of the mixture passing therethrough. By way of example, heating sections 26 made from a 17.8 mm (0.7 inch) inner diameter transparent tempered glass tubes having a length of 152.4 mm (6 inches) supplied by QVF Process Systems, Inc., Horseheads, New York are well suited for use in the present  
15 invention. If no visual inspection is needed, then heating section 26 may be made of chemically inert material, such as steel, and stainless steel.

Details of heating zone 28 can be seen FIGS. 2A, 2B and 2C. Heating zone 28 includes a flange 286 provided with a heat transfer fluid intake 281 and drain 282. A typical heat transfer fluid used is steam. However, other heating fluids, included heated  
20 gas are also contemplated for use in the present invention. A steam pocket 293 within flange 286 is sealed by conventional sealing means, such as flange 'O' ring 294, contacted against a flange cover 284 to prevent leakage of the heat transfer fluid, which is typically supplied at high temperature and pressure. When steam is used as the fluid, steam is supplied at a temperature and pressure, respectively in the range from 20 KPa at  
25 60°C to 22 MPa at 374°C, preferably from 101 KPa at 100°C to 4 MPa at 250°C. Typically securing means, such as screws 298, shown in FIG. 2A are used to secure flange cover 284 against flange 286. Heating sections 26 are sealably anchored to flange 286 and flange cover 284 by clamp assembly 291. Clamp assembly 291 includes a gasket insert 288, a sealing gasket 290 and a wrap around metal clamp 292. Gasket insert 288  
30 typically made from a heat insulating material, such as Teflon<sup>®</sup> tetrafluoroethylene, supplied DuPont Company, Wilmington, Delaware is interposed between centrally disposed openings on flange 286 and flange cover 284 to prevent transfer of heat from heating zone 28 to heating sections 26. Sealing gasket 290 made from a flexible material,

such as silicon rubber is mounted over gasket insert 288 and substantially identical lips provided on heating sections 26 and the openings on flange 286 and flange cover 284. Clamp 292 is mounted over sealing gasket 290 to sealably secure heating sections 26 to heating zone 28.

Centrally disposed tubular projection on flange cover 284 abutting against a mating tubular projection on flange 286 is sealed with an 'O' ring seal 296 to allow a plug free passage of the mixture through means 5 for heating. As shown in Figs. 2C, 2D and 2E, two or more injection ports 299 are preferably provided on the centrally disposed tubular projection on flange cover 284 to permit injection of heat transfer fluid supplied to heating zone 28. The orifice of injection port 299 is kept small enough to prevent cavitation (formation of vapor bubbles) in heating zones 28. As shown in FIGS. 2D and 2E, injection ports 299 are preferably positioned opposite to one another on the centrally disposed tubular projection on flange cover 284, such that the injected fluid from one port 299 impinges against the injected fluid from the opposite port 299. Ports 299 are offset to produce a swirl pattern within the mixture to improve the heat transfer from the heat transfer fluid to the polymer pellets. Alternatively, ports 299 can be positioned diametrically opposite to one another such that the injected fluid from one port 299 directly impinges against the injected fluid from the opposite port 299. It is well within the scope of this invention to provide a single port 299 or a plurality of ports 299, such as, for example, 3 or 4 ports positioned at 180 degrees from each other, or at 90 degrees from each other, respectively.

To further improve the heat transfer from the heat transfer fluid to the polymer pellets, the swirl pattern created in adjacent heating zones 28 may be reversed at some places or alternated thorough out means 5 for heating to further improve the heart transfer. Thus, if a clockwise swirl pattern is created in one heating zone 28, ports 299 on adjacent flange covers 284 in adjacent heating zone 28 could be repositioned by up to 180° to produce a counter-clockwise swirl pattern.

Means 5 for heating may be positioned vertically (as a column) or horizontally. A horizontal configuration is preferred, since the plug flow condition can be achieved at lower transportation velocity of the mixture passing through the heating means than that obtained in a column configuration. Preferably, heating means 5 is inclined to accumulate a pocket of air in the mixture passing through heating means 5 from pump

means 22 towards shearing means 7 described below, which can then be vented out during start up. The end of means for heating 5 proximate to metering pump means 22 is positioned 1 to 10 degrees higher than the end of means 5 for heating proximate to means

5 7 for shearing.

The length of heating means 5 resulting from linearly securing a plurality of heating sections 26 and heating zones 28 is adjusted to heat the core of the pellets to a desired temperature above the melting temperature or glass transition temperature.

Generally, the polymer pellets are heated above the melting temperature of the polymer.

10 The time required to heat the core of the pellet can be calculated by using the following equation:

$$\frac{T_1 - T}{T_1 - T_0} = 2 \sum_{n=0}^{\infty} \frac{(-1)^n}{(n + 1/2)\pi} e^{-\left(n + \frac{1}{2}\right)^2 \pi^2 \alpha / r^2} \cos\left(n + \frac{1}{2}\right) \frac{\pi r}{R}$$

where r is the radius of the core of the pellet, R is the radius of the pellet, T is the temperature at radius r, T<sub>0</sub> is the temperature of the medium, T<sub>1</sub> is the initial temperature of the pellet, and α is the thermal diffusivity of the polymer.

Based on the requirement necessary to satisfy the minimum transportation velocity, a residence time vs. length was estimated. The residence time was then checked against the results from the equation above. Based on these calculations the largest pellet size that can be used for heating sections 26 sizes described below is equal or less than 3 mm (1/8 inch). Therefore, a good balance can be achieved with a plurality of glass heating sections 26 each having an inner diameter of 17.8 mm at an over all length of 3.05 meter (10 feet) and metering pump means 22 supplying the mixture passing through heating means 5 at a flow rate in the range of 2.84 to 7.6 liters per minute (0.75 to 2 gallons per minute) to heat the core of a 3 mm (1/8 inch) pellets to within 95% of the temperature of the mixture.

The temperature of the mixture, heat transfer fluid supplied to the mixture and the heating medium are monitored by means of conventional thermocouples (not shown) to control the temperature of the mixture. The pressure and the flow rate of heat transfer fluid supplied to heating zones 28 is also monitored and controlled by conventional

means. If desired, the entire means 5 for heating can be enclosed in an enclosure, preferably a transparent enclosure, for safety.

An output port 30 exiting from heating means 5 enters shearing means 7, which include one or more conventional shear devices 32. One suitable shearing device supplied by E. T. Oakes Corporation, Hauppauge, New York, Model No. 4MB1A is particularly preferred. However, applicants contemplate using a variety of other suitable shear devices to shear the heated polymer pellets supplied from heating means 5 into polymer particles of desired size, sphericity and particle size distribution.

If desired, one or more additional components, such as a catalyst, surfactant or any other additives, can be introduced at a desired rate via input port 34 positioned on an output passage 36 exiting from shearing device 32 or at output port 30 exiting from heating means 5. As a result, catalyst can thus come in contact with sheared polymer particles before they are cooled, preferably to room temperature by utilizing cooling means 9. However, applicants contemplate suitably positioning one or more ports anywhere in device 1 to introduce one or more additional components.

Cooling means 9 include an elongated cooling passage 38, preferably a metallic tube, connected to output passage 36 and free from any substantial obstruction to permit passage of the mixture therethrough under the plug free conditions. A cooling jacket 40 surrounds cooling passage 38 supplied with a conventional coolant, such as water, a blend of water and ethylene glycol, entering from a port 42 and exiting from a port 45 thereby providing a cross-convexional heat transfer for improved heat transfer efficiency. The temperature and the rate at which the coolant is supplied are monitored and controlled by conventional means (not shown) to attain desired cooling rates. Means 9 for cooling may be optionally provided with an axially disposed agitator to prevent the polymer particles from settling down during the passage through cooling means 9.

Means 9 for cooling may be positioned vertically (as a column) or horizontally. Horizontal configuration is preferred, since the plug flow condition can be achieved at lower transportation velocity of the mixture passing through heating means 5 than that obtained in a column configuration. Preferably, means 9 for cooling are inclined to accumulate air in the mixture passing through means 9 for cooling from shearing means 7 towards passage 44, which then can be vented out during the start up. The end of means

9 for cooling proximate to shearing means 7 is positioned 1 to 10 degrees lower than the end of means 9 for cooling proximate to passage 44.

In order to prevent cavitation either in heating means 5 or in cooling means 9, it is desirable that the operating pressure of the process is maintained at a pressure that prevents boiling of the individual components in the mixture or the mixture itself. The mixture containing the polymer particles can be removed from device 1 in various ways while still maintaining the operating pressure that prevents cavitation. One of the ways of reducing the operating pressure exiting from means 9 for cooling to atmospheric pressure is shown in FIG. 1A. A delivery port 52 of pressure let-down pump means 50 is connected to an exit passage 44 to provide a back pressure against the operating pressure exiting from exit passage 44. Thus, by monitoring the exit pressure from exit passage 44, the back pressure provided by pressure let-down pump means 50 can be constantly adjusted to maintain the pressure in a process output passage 46 at atmospheric pressure. The mixture containing the polymer particles can then be stored in a storage tank 48. If desired, additional water may be added via port 54 to storage tank 48.

The mixture containing the polymer particles, also known as aqueous slurry can be applied conventionally, by such means, spraying, dipping and roller coating, over a wide variety of substrate surfaces.

In addition to the detailed description of the preferred embodiment provided herein, it should be understood that the present invention may be embodied in other various forms.

If desired, device 1 of the present invention can include means for producing a powder of polymer particles obtained by evaporating the aqueous medium from the aqueous slurry. As shown in FIG. 1A, the aqueous slurry is conveyed through an exit passage 58 to an evaporation chamber 56. The aqueous slurry is flashed through a flash nozzle 60 within chamber 56 for removing the volatiles via an exhaust port 62. Evaporation chamber 56 is preferably maintained under vacuum for removing the volatiles from the aqueous slurry. Evaporation chamber 56 is provided with a conventional exit port 66 to remove polymer particles 64 produced in a powder form from evaporation chamber 56.

Applicants contemplate a variety of alternate embodiments to the aforescribed embodiment. For example, a capillary tube may be substituted for pressure let-down pump means 50 to bring the operating pressure down to atmospheric pressure.

5 An alternative embodiment 9A to cooling means 9 is shown in Figs. 3A, 3B and 3C. Means 9A include co-axially coiled outer and inner tubes 904 and 906, respectively coiled to increase the cooling surface without increasing the space required for cooling means 9A. An insulator 902 covers the outer surface of tube 904 to substantially insulate cooling medium passing between outer tube 904 and inner tube 906. For improved  
10 cooling efficiency coolant flows in opposite direction to the flow of the mixture passing through inner tube 906.

If desired and depending upon the chemical make-up and size of the polymer pellets, and the viscosity of pellets being sheared under molten conditions, additional shear devices may be used instead of a single shear device 32. It is further contemplated  
15 that the sheared particles existing from the first shear device may be reheated again through another set of heating means, similar to heating means 5, before re-shearing the polymer particles through a second set of a shear device.

Even though steam as a heating fluid is preferred, it is within the purview of this invention to contemplate other water soluble heat transfer fluids, such as methyl ethyl  
20 ketone, methyl amyl ketone, acetone, ethylene glycol or blends thereof. However, it is essential that such fluids do not affect the plug flow of the mixture. It is also contemplated that the mixture may contain miscible solvents or that the aqueous medium may be substituted with a non-aqueous liquid medium having boiling point higher than the melting point of the polymer pellets.

25 In operation, the process of the present invention, step (i) for producing substantially round polymer particles includes forming a mixture of the polymer pellets, at least one surfactant and an aqueous medium in slurry tank 3. Preferably, the pellets are supplied from pellet hopper 12. The aqueous medium includes water, preferably deionized water. If desired and depending on the chemical make-up of polymer pellets  
30 being sheared, one or more miscible co-solvents, such as primary, secondary and tertiary alcohols, methyl ethyl ketone, and methyl amyl ketone may also be included in aqueous medium. The selection of the surfactant used typically depends upon the chemical make-up of the polymer pellets being sheared. As stated earlier, the surfactants can be blended

in with the polymer to form the polymer pellets before the pellets are conveyed to slurry tank 10. Generally, the surfactant is selected from at least one member of the group consisting of ionic, nonionic surfactants and a combination thereof. Preferred ionic surfactants are those formed in situ by the reaction of one or more of ammonium hydroxide, triethanolamine, morpholine and dimethyl ethanolamine with the carboxyl functionality integral to the copolymer. Other suitable ionic surfactants include ammonium lauryl sulfate, sodium lauryl sulfate and sodium dodecyl benzene sulfonate. Sodium lauryl sulfate and sodium dodecyl benzene sulfonate are particularly preferred with thermosetting polymers, such as, for example, polyester-epoxy hybrids and glycidyl methacrylates with 1,12-dodecanedioic acid. Preferred nonionic surfactants are polyoxypropylene-polyoxyethylene block copolymer (Pluronic® F-98 supplied by BASF Performance Chemicals in Mount Olive, New Jersey), alkylphenol thioxylates, and ethyleneoxide-propylene glycol polymers. Nonionic surfactants having a cloud point in the range of 100°C to 150°C are particularly preferred. The cloud point is defined as the temperature at which a surfactant begins to dehydrate and self-associate rather than remain soluble in water. The surfactant concentration should be sufficient to prevent coalescence of polymer particles formed in means 7 for shearing, described below. Generally, the surfactant concentration in the range of about 0.001% to 25% by weight of the polymer pellets, preferably in the range of about 0.01% to 5% of the ionic surfactant is added to the mixture. In addition, in the range of about 0.001% to 25% by weight of the polymer pellets, preferably in the range of about 0.1% to 10% of the nonionic surfactant can be added to the mixture.

Typically, one or more other conventional components, such as, for example, a pigment (metallic and non-metallic pigments), dye, anticorrosion agent, anti-foaming agent, catalyst, ultraviolet light (UV) stabilizer, UV absorber, antioxidant, flow modifier, degassing aid, filler, extender, gloss-reducing additive, texturizing aid, abrasion resisting additive, anti-microbial agent, biocide, fungicide, photo-initiator, pH modifier, thorough-cure modifier, color stabilizer or a leveling agent can be also included in the mixture during the step (i). Alternatively, the aforescribed components can be blended in the polymer pellets themselves or can be conveniently introduced at any point in device 1.

The polymer pellets suitable for use in the present invention include one or more conventional thermoplastic or thermosetting polymers that may be crystalline, semi-

crystalline or amorphous. If desired a blend of one or more thermoplastic and/or one or more thermosetting polymers can be also used.

The polymer pellets may be in any form including but not limited to powder, flake  
 5 or chopped extrudate. Since the pellets are heated in the heating step described below, applicants have found that when heating means 5 having the dimensions described herein are used, the core of polymer pellets having less than 3 mm in size can be heated more readily during the heating step than the core of pellets of a larger size. However, as described earlier, the upper limit of the polymer pellet size depends upon the inner  
 10 diameter of heating sections 26. Thus, the larger the inner diameter of heating section 26, the higher can be the upper limit of the polymer pellet size. Generally, the inner diameter of heating sections 26 is about 8 to 10 times the size of the polymer pellet being heated. The polymer pellet concentration can range from about 0.5 to 50% by volume, based on the volume of the mixture. A polymer concentration below 40%, more preferably 10% to  
 15 35%, is preferred since the viscosity of the aqueous slurry increases rapidly as the concentration of polymer pellet approaches that at which the mixture would behave as a wet cake rather than a liquid.

Some of the suitable thermoplastic polymer and copolymer that can be used in this invention are disclosed in U.S. Pat. Nos. 3,933,954, 3,264,272 and Canadian Pat. No.  
 20 655,298, all of which are hereby incorporated by reference. The suitable polymers are copolymers of  $\alpha$ -olefins of the formula  $R-CH=CH_2$ , where R is hydrogen or an alkyl radical of 1 to 8 carbon atoms; and  $\alpha$ ,  $\beta$ -ethylenically unsaturated carboxylic acids having from 3 to 8 carbon atoms; and, optionally, a monoethylenically unsaturated monomer. Suitable olefins include ethylene, propylene, butene-1, pentene-1, hexene-1, heptene-1, 3-  
 25 methylbutene-1, and 4-methylpentene-1. Examples of  $\alpha$ ,  $\beta$ -ethylenically unsaturated carboxylic acids are acrylic acid, methacrylic acid, ethacrylic acid, itaconic acid, maleic acid, fumaric acid, monoesters of the dicarboxylic acids, such as methyl hydrogen maleate, methyl hydrogen fumarate, ethyl hydrogen fumarate and maleic anhydride. Although maleic anhydride is not a carboxylic acid in that it has no hydrogen attached to  
 30 the carboxyl groups, it can be considered an acid for the purposes of the present invention because of its chemical reactivity being that of an acid. Similarly, other  $\alpha$ ,  $\beta$ -monoethylenically unsaturated anhydrides of carboxylic acids can be employed. The preferred unsaturated carboxylic acids are methacrylic and acrylic acids. As indicated,



the concentration of acidic monomer in the copolymer is from 0.2 mol percent to 25 mol percent, and, preferably, from 1 to 10 mol percent.

The copolymer base need not necessarily comprise a two-component polymer.

- 5 Thus, although the olefin content of the copolymer should be at least 50 mol percent, more than one olefin can be employed to provide the hydrocarbon nature of the copolymer base. Additionally, other copolymerizable monoethylenically unsaturated monomers, mentioned in the following paragraph, can be employed in combination with the olefin and the carboxylic acid comonomer. The scope of base copolymers suitable for  
10 use in the present invention is illustrated by the following two component examples:

Ethylene/acrylic acid copolymers, ethylene/methacrylic acid copolymers, ethylene/itaconic acid copolymers, ethylene/methyl hydrogen maleate copolymers, and ethylene/maleic acid copolymers. Examples of tricomponent copolymers include:  
15 ethylene/acrylic acid/methyl methacrylate copolymers, ethylene/methacrylic acid/ethyl acrylate copolymers, ethylene/itaconic acid/methyl methacrylate copolymers, ethylene/methyl hydrogen maleate/ethyl acrylate copolymers, ethylene/methacrylic acid/vinyl acetate copolymers, ethylene/acrylic acid/vinyl alcohol copolymers, ethylene/propylene/acrylic acid copolymers, ethylene/styrene/acrylic acid copolymers, ethylene/methacrylic acid/acrylonitrile copolymers, ethylene/fumaric acid/vinyl methyl  
20 ether copolymers, ethylene/vinyl chloride/acrylic acid copolymers, ethylene/vinylidene chloride/acrylic acid copolymers, ethylene/vinyl fluoride/methacrylic acid copolymers, and ethylene/chlorotrifluoroethylene/methacrylic acid copolymers.

In addition to the third monomer component of the ionic copolymer stated above, additional third monomeric components can be an alkyl ester of an  $\alpha,\beta$ -ethylenically  
25 unsaturated carboxylic acid of 3 to 8 carbon atoms where the alkyl radical has 4 to 18 carbon atoms. Particularly preferred are the terpolymers obtained from the copolymerization of ethylene, methacrylic acid, and alkyl esters of methacrylic acid or acrylic acid with butanol. The concentration of this optional component is 0.2 mol to 25 mol percent, based on the weight of copolymer, preferably from 1 mol to 10 mol percent.

30 Representative examples of the third component include n-butyl acrylate, isobutyl acrylate, secondary butyl acrylate, tertiary butyl acrylate, n-butyl methacrylate, isobutyl methacrylate, sec-butyl methacrylate, t-butyl methacrylate, n-pentyl acrylate, n-pentyl methacrylate, isopentyl acrylate, isopentyl methacrylate, n-hexyl acrylate, n-hexyl

methacrylate, 2-ethyl-hexyl acrylate, 2-ethyl-hexyl methacrylate, stearyl acrylate, stearyl methacrylate, n-butyl ethacrylate, 2-ethyl hexyl ethacrylate. Also, the third component includes mono- and di-esters of 4 to 8 carbon atom dicarboxylic acids such as n-butyl

5 hydrogen maleate, sec-butyl hydrogen maleate, isobutyl hydrogen maleate, t-butyl hydrogen maleate, 2-ethyl hexyl hydrogen maleate, stearyl hydrogen maleate, n-butyl hydrogen fumarate, sec-butyl hydrogen fumarate, isobutyl hydrogen fumarate, t-butyl hydrogen fumarate, 2-ethyl hexyl hydrogen fumarate, stearyl hydrogen fumarate, n-butyl fumarate, sec-butyl fumarate, isobutyl fumarate, t-butyl fumarate, 2-ethyl hexyl fumarate,

10 stearyl fumarate, n-butyl maleate, sec-butyl maleate, isobutyl maleate, t-butyl maleate, 2-ethyl hexyl maleate, stearyl maleate. The preferred alkyl esters contain alkyl groups of 4 to 8 carbon atoms. The most preferred contain 4 carbon atoms. Representative examples of the most preferred esters are n-butyl acrylate, isobutyl acrylate, butyl methacrylate, isobutyl methacrylate, tertiary butyl acrylate, tertiary butyl methacrylate.

15 The copolymers may also, after polymerization but prior to ionic crosslinking, be further modified by various reactions to result in polymer modifications, which do not interfere with the ionic crosslinking. Halogenation of an olefin acid copolymer is an example of such polymer modification.

The preferred base copolymers, however, are those obtained by the direct

20 copolymerization of ethylene with a monocarboxylic acid comonomer.

Metal ions, which are suitable in forming the ionic copolymers of the present invention, can be divided into two categories, uncomplexed metal ions and complexed metal ions. In the uncomplexed metal ions the valence of the ion corresponds to the valence of the metal. These metal ions are obtained from the commonly known and used

25 metal salts. The complexed metal ions are those in which the metal is bonded to more than one type of salt group, at least one of which is ionized and at least one of which is not. Since the formation of the ionic copolymers requires only one ionized valence state, it will be apparent that such complexed metal ions are equally well suited in the present invention. The term "metal ion having one or more ionized valence states" means a metal

30 ion having the general formula  $Me_n X_m$ , where n is the ionic charge and is at least one, X is a non-ionized groups and  $n + m$  equal the valence of the metal. The utility of complexed metal ions employed in the formation of ionic copolymers corresponds in their ionized valences to those of the uncomplexed metal ions. The monovalent metals are, of

course, excluded from the class of complexed metal ions but higher valent metals may be included depending on how many metal valences are complexed and how many can be ionized. The preferred complexed metal ions are those in which all but one metal

5 valences are complexed and one is readily ionized. Such compounds are in particular the mixed salts of very weak acids, such as oleic and stearic acid, with ionizable acids, such as formic and acetic acid.

The uncomplexed metal ions which are suitable in forming the ionic copolymers of the present invention, therefore comprise for the  $\alpha$ -olefin-monomonocarboxylic acid

10 copolymers, mono-, di- and trivalent ions of metals in Groups I, II, III, IV-A and VIII of the Periodic Table of Elements (see page 392, Handbook of Chemistry and Physics, Chemical Rubber Publishing Co., 37th ed.). Uncomplexed monovalent metal ions of the metals in the stated groups are also suitable in forming the ionic copolymers of the present invention with copolymers of olefins and ethylenically unsaturated dicarboxylic

15 acids. Suitable monovalent metal ions are  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Li}^+$ ,  $\text{Cs}^+$ ,  $\text{Ag}^+$ ,  $\text{Hg}^+$  and  $\text{Cu}^+$ . Suitable divalent metal ions are  $\text{Be}^{+2}$ ,  $\text{Mg}^{+2}$ ,  $\text{Ca}^{+2}$ ,  $\text{Sr}^{+2}$ ,  $\text{Ba}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Hg}^{+2}$ ,  $\text{Sn}^{+2}$ ,  $\text{Pb}^{+2}$ ,  $\text{Fe}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Ni}^{+2}$  and  $\text{Zn}^{+2}$ . Suitable trivalent metal ions are  $\text{Al}^{+3}$ ,  $\text{Sc}^{+3}$ ,  $\text{Fe}^{+3}$  and  $\text{Y}^{+3}$ .

The preferred metals, regardless of the nature of the base copolymer, are  $\text{Na}^+$  and  $\text{Zn}^{+2}$ . These metals are preferred because they result in ionic copolymers having the best

20 combination of improvement in solid state properties with retention of melt fabricability. It is not essential that only one metal ion be employed in the formation of the ionic copolymers and more than one metal ion may be preferred in certain applications.

The thermosetting polymer pellets suitable for use in the present invention preferably incorporate crosslinking agents with polymers or copolymers having

25 crosslinkable functionalities, such that, upon application, when the functionalities from a crosslinking agent crosslink with those on the polymer, coatings having additional benefits, such as improved in corrosion resistance, adhesion and mechanical properties, are produced. Several techniques have been disclosed to pre-blend the crosslinking agents with polymers for producing thermosetting polymer pellets. For example,

30 reactivity and process conditions must be such that substantially no crosslinking occurs during the pellet formation process. Generally, the crosslinking agent is dispersed or solubilized in the mixture containing the thermosetting polymer pellets.

Some of the most suitable crosslinkable functionalities on a polymer used in the thermosetting polymer pellets include those that can be thermally crosslinked, actinically crosslinked, or a combination thereof. Some the suitable crosslinkable functionalities include acid or anhydride functionalities. Suitable crosslinking agents include, but are not limited to hydroxyalkylamide and epoxy containing materials, such as diglycidyl ether of bisphenol A, diglycidyl ether of bisphenol F and triglycidyl isocyanurate.

Optionally an additional monomer with hydroxyl functionality, such as, but not limited to, hydroxyethyl acrylate, hydroxyethylmethacrylate or hydroxypropyl methacrylate, can be incorporated into the polymer. Suitable crosslinking agents that can be incorporated into the particles include blocked or free isophorone diisocyanate, blocked toluene diisocyanate and blocked or free tetramethylene diisocyanate. Caprolactone is a commonly used blocking agent. Other crosslinkers known in the art that can be used, include aminoplast crosslinking resin, such as, but not limited to, tetramethoxymethyl glycouril.

Typically, the thermosetting polymer pellets include a crosslinking agent in the stoichiometric range of about 1:0.05 to 1:1.5 with respect to the acid group in the crosslinkable polymer. Preferably, the range is between 1:0.1 to 1:1. The range of acid functionality in the crosslinkable polymer before crosslinking is between about 1 and 30 percent by weight of the ethylene. Preferably, the range is between 4 to 20 percent. The hydroxy functionality in the crosslinkable polymer, when present, before crosslinking is between about 0.001 to 30 percent by weight of the ethylene. Preferably, the range is between 2 to 10 percent.

Step (ii) of the process of the present invention includes heating the polymer pellets in means 5 for heating as the mixture is conveyed under plug flow and plug free conditions through heating means 5. The plug flow plug free conditions are attained in heating means 5 by maintaining the Reynolds Number of the mixture passing through mean for heating 5 at about 2,000 to 500,000, preferably 5,000 to 200,000 and most preferably 6000 to 150,000; and by maintaining the transportation velocity of the polymer pellets in means 5 for heating at or above the Durand's minimum transportation velocity expressed by the following equation:

$$V_{MH} = F_1 \left[ 2gD_H \left( \frac{\Delta \rho}{\rho_{medium}} \right) \right]^{0.5}$$

where  $V_{MH}$  is minimum transportation velocity of the mixture,  $F_1$  is an empirically derived constant that depends on the size of the polymer pellet being conveyed,  $g$  is the acceleration due to gravity,  $D_H$  is the inner diameter of heating sections 26,  $\Delta \rho$  is the absolute value of the difference between the densities of the polymer pellets and the aqueous medium and  $\rho_{medium}$  is the density of the aqueous medium.

The polymer pellets are melted by the heat transfer fluid, preferably steam under pressure supplied through intake 281 on each of the plurality of heating zones 28. The condensed steam is readily drained from drain 282 in heating zone 28. The temperature of the mixture containing the pellets is raised in steps by the plurality of heating zones 28 as the medium passes from one heating section 26 to the next heating section 26. Depending upon the chemical make up of the pellets, the temperature is raised sufficiently rapidly to permit efficient shearing of the pellets. Preferably, the pellets are heated above  $T_g$  in case of amorphous polymers and above  $T_m$  for crystalline and many of the semi-crystalline polymers. Thus, as the pellets exit from means 5 for heating, they are substantially in a molten state. Depending upon the chemical make up of the polymer pellets, the pellets attain temperatures above the melting point or glass transition temperature. By way of example, when Nucrel® RX-76 polymer pellets supplied by DuPont Company of Wilmington, Delaware, were heated to temperature in the range of 110°C to 150°C. The residence time of the polymer pellets in means 5 for heating was about 10 to 2 seconds.

A time/temperature profile is preferably utilized during the heating or cooling polymer pellets. By "time/temperature profile" it is meant that lower temperatures can be employed for longer times and higher temperatures for shorter times, all within the temperature ranges and times discussed herein. The balance of time/temperature is important to avoid significant (more than, say, about 35% percent) reaction of the crosslink agent with the copolymer before such crosslinking is desired, which is after application of the particles to the substrate to be coated. The rate at which the temperature is raised during the residence time of the pellets in means 5 for heating

depends upon the type of polymer being heated. Thermosetting polymer particles blended with crosslinkers would be highly temperature sensitive as they can prematurely crosslink if exposed to temperatures sufficiently high and a residence time sufficiently long to result in premature crosslinking of more than 35%. Generally, the conditions are adjusted to ensure that any premature crosslinking is no more than about 0.5% to 35%, preferably no more than about 0.1% to 25% and most preferably no more than 0.05% and 10%.

In the next step (iii), melted pellets in the heated mixture exiting from output port 30 enter shearing means 7, which include one or more conventional shear devices 32. The melted polymer pellets are sheared into polymer particles having a desired sphericity, particle size distribution and size range. Typically, shear rates in the range of from about 1000 to 32,000 1/s are employed. When more than one shearing is used, the exiting polymer particles from the first shearing device are preferably reheated to a desired temperature before the reheated particles enter the next shearing device. The size of polymer particles obtained are controlled conventionally, such as by the type of shearing device used, the rate at which the polymer pellets are sheared and the temperatures of the pellets being sheared and the amount of the surfactant added to the mixture. Thus, for the same pellet loading, i.e., amount of pellets added to the mixture, the higher the shear rate, generally the smaller will be the average particle size.

For more difficult to shear polymer particles, such as Nucrel® 960 polymer supplied by DuPont Company, Wilmington, Delaware, shearing is preferably done in stages by utilizing more than one shear device 32.

Most practical particle size ranges when applied over substrate surfaces in the form of an aqueous slurry are 100 to 10,000 nm, preferably 150 to 6,000 nm, especially preferred 400 to 4,000 nm, and highly preferred 800 to 3,500 nm. Most practical particle size ranges when applied over substrate surfaces in the form of a powder are 100 to 1,000,000 nm, preferably 500 nm to 500,000 nm, especially preferred 1000 to 250,000 nm, highly preferred 2000 to 100,000 nm.

If desired, a desired amount of catalyst can be added through port 34 to the mixture after the melted pellets are sheared. If more than one shearing device 32 is utilized, then the addition of catalyst preferably takes place before the pellets being

sheared enter the last shearing device 32. Alternatively, a desired amount of catalyst can be added to the mixture before the melted pellets are sheared.

While a neutralization step is not needed when thermosetting polymers are utilized, it is generally employed when converting ethylene/methacrylic acid polymers to the corresponding ionomers. Some of the suitable neutralizing agents, added through port 54 to control the pH of the aqueous medium, include amines, ammonia, zinc carbonate and metal hydroxides, such as sodium hydroxide. Ammonia, sodium hydroxide, potassium hydroxide and zinc carbonate are preferred.

During (iv) step, the polymer particles exiting from means 7 for shearing are then cooled under plug free conditions through means 9 for cooling. Generally, cooling to room temperature is accomplished fairly rapidly. The rate of cooling, the incoming temperature of the coolant being used is dependent on the chemical make up of polymer particles being cooled. Thus, for example, when temperature sensitive blend of particles containing a thermosetting polymer and a crosslinking agent are sheared, it is preferable to cool them under plug flow cooling conditions to ensure minimal degree of crosslinking, defined earlier. The use of plug flow conditions in cooling thermoplastic polymer particles is also preferred.

When plug flow conditions are utilized in means 9 for cooling; the Reynolds Number of the mixture passing through means 9 for cooling is maintained in the range of about 2,000 to 500,000, preferably 5,000 to 200,000 and most preferably 6000 to 150,000.

Additionally, the transportation velocity of the polymer particles in means 9 for cooling is maintained at or above the Durand's minimum transportation velocity expressed by the following equation:

$$V_{MC} = F_2 \left[ 2gD_C \left( \frac{\Delta \rho}{\rho_{medium}} \right) \right]^{0.5}$$

where  $V_{MC}$  is minimum transportation velocity of the mixture,  $F_2$  is an empirically derived constant that depends on the size of the polymer pellet being conveyed,  $g$  is the acceleration due to gravity,  $D_C$  is the inner diameter of cooling passage 38 of means 9 for cooling,  $\Delta \rho$  is the absolute value of the difference between the densities of the polymer pellets and the aqueous medium and  $\rho_{medium}$  is the density of the aqueous medium.

As stated earlier, to prevent cavitation either in heating means 5 or in cooling means 9, the pressure of the mixture exiting from means 9 for cooling is reduced to atmospheric pressure from the operating pressure by means, such as let-down pump means 50 connected to an exit passage 44 or by means of capillary tube of a known length. The mixture containing the polymer particles is then stored in a storage tank 48. If desired, additional water may be added via port 54 to storage tank 48.

If desired, the polymer particles from the aqueous slurry may be isolated by any of the well-known methods, including but not limited to decantation, drying, centrifugation, or filtration. FIG. 1A illustrates one such conventional approach where the aqueous slurry is flashed in evaporation chamber 56 through flash nozzle 60 to remove the volatiles via exhaust port 62. The polymer particles 64 in a powder form can then be conventionally removed via exit port 66. Generally, evaporation takes place at sub-atmospheric pressures in the range typically used in such processes.

### **EXAMPLES**

Unless otherwise specified, all chemicals and reagents were used as received from Sigma-Aldrich, St. Louis, MO.



### Thermoplastic Materials

The following procedure was used to produce polymer particles of Example A:

5       “NUCREL® 960” ethylene methacrylic acid copolymer (supplied by DuPont Company, Wilmington, Delaware) polymer pellets (thermoplastic polymer density of 0.95 g/cc and melting point of 95°C) having pellet size of about 3 mms were simultaneously fed with agitation into slurry tank 10 with deionized water and surfactant (Pluronic® F-98 polyoxypropylene-polyoxyethylene block copolymer supplied by BASF

10   Performance Chemicals in Mount Olive, New Jersey. Cloud point of about 107°C). The loading of the polymer pellets in the mixture was at 10 weight parts per hundred (PPH) and that of the surfactant was at 1 (PPH). The temperature in slurry tank 10 was maintained 28°C. The mixture was then heated through heating means 5 to heat the polymer pellets under plug flow conditions. The temperature of the mixture was heated

15   by steam at 150°C supplied at 618 kiloPascals (75 psig) system pressure. As a result, the temperature of the mixture at output port 30 was 135°C. Metering pump means 22 supplied pressure at 653 kiloPascals (80 psig) at motor RPM of 850. The residence time in heating means 5 was 9.88 seconds. The mixture exiting from output port 30 was sheared in shearing device 32 operating at 1065 RPM. The shear rate was maintained at

20   7,645 1/s with a shear residence time of 1.43 sec. The mixture containing sheared polymer particles that exited from output passage 38 was then cooled in an agitated cooling passage 40 to a temperature of about 24°C. The coolant was supplied at temperature of 6°C. Motor RPM of pressure let down pump means 50 was 600. The flow rate the mixture at delivery port 24 was 0.0000669 meters<sup>3</sup> per second (1.06 GPM)

25   and the flow rate of the mixture exiting out of exit port 44 was 0.0000959 meters<sup>3</sup> per second (1.52 GPM). The increase in the GPM rate occurred due to the condensation of steam added in heating zones 28. Initial velocity of the mixture at delivery port 24 was measured 0.22 meters per second (0.72 feet per second) and the final velocity of the mixture at output port 30 was measured 0.31 meters per second (1.03 feet per second).

30   Additionally, Durand’s Velocity was determined to be 0.19 meters per second (0.63 feet per second), and as described in the discussion on the plug flow conditions above, this

process was determined to be in compliance with the plug flow definition. Rounded polymer particles of an approximate size of less than 100  $\mu$  were isolated.

Examples B and C were made using the process as described above. "NUCREL® RX76", supplied by DuPont, Wilmington, DE was used in Example B. Low-density polyethylene (LDPE, RIBLENE® MV 10, supplied by Polimeri Europa, Milano, Italy) was used in Example C. The results of Examples A, B and C are summarized in Table 1 below.

### Thermoset Materials

Examples D, E, F and G, summarized in Table 1 below, were done using thermoset materials as described herein.

In Example D, thermosetting polymer pellets were prepared by mixing 100 PPH of Nucrel® RX-76 ethylene methacrylic acid copolymer supplied by DuPont Company, Wilmington, Delaware with 40 PPH of ethylene glycidyl methacrylate crosslinking agent supplied by DuPont Company, Wilmington, Delaware and then extruder pelletized at 109°C in a 40 mm extruder. The pellet size was in the range of 3 mm and a polymer density of 0.95 g/cc. The processing temperature of the polymer blend was determined by measuring its dynamic viscosity, which was constant (about 100 Pascal-Second) at 130°C indicating substantially no crosslinking taking place at that temperature, whereas the dynamic viscosity at 180°C increased rapidly, indicating the presence of crosslinking taking place. Thus, it can be seen that the thermosetting polymer pellet could be safely processed at less than 130°C without producing substantially premature crosslinked polymer particles.

The thermosetting polymer pellets described above were simultaneously fed with agitation into slurry tank 10 with deionized water and surfactant (Pluronic® F-98 polyoxypropylene-polyoxyethylene block copolymer supplied by BASF Performance Chemicals in Mount Olive, New Jersey). The loading of the polymer pellets in the mixture was at 5 weight parts per hundred (PPH) and that of the surfactant was at 1 (PPH). The temperature in slurry tank 10 was maintained 28°C. The mixture was then conveyed into means 5 for heating to heat the polymer pellets under plug flow conditions. The temperature of the mixture was heated by steam supplied at 150°C supplied at 618 kiloPascals (75 psig) system pressure. As a result, the temperature of the mixture at

output port 30 was 128°C. Metering pump means 22 supplied pressure at 639 kiloPascals (78 psig) at motor 420 RPM. The residence time in heating means 5 was 9.69 seconds. The mixture exiting from output port 30 were sheared in shearing device 32 operating at 500 RPM. The shear rate was maintained at 3,589 1/s with a shear residence time of 1.67 sec. The mixture containing sheared polymer particles that exited from output passage 36 was then cooled in a plug flow cooling passage shown in FIG. 3A to a temperature of about 18°C. The coolant was supplied at temperature of 6°C. Motor RPM of pressure let down pump means 50 was 520. The flow rate of the mixture at delivery port 24 was 0.00006309 meters<sup>3</sup> per second (1.0 GPM) and the flow rate of the mixture exiting out of exit port 44 was 0.0000820 meters<sup>3</sup> per second (1.30 GPM). The increase in the GPM rate occurred due to the condensation of steam added in heating zones 28. Initial velocity of the mixture at delivery port 24 was measured 0.22 meters per second and the final velocity of the mixture at output port 30 was measured 0.29 meters per second. Additionally, Durand's Velocity was determined to be 0.18 meters per second. The process, as described in the discussion on the plug flow conditions above, was determined to be in compliance with the plug flow definition. Rounded polymer particles of an approximate size of 10-50 µm were isolated.

Example E was prepared in a similar manner to Example D. To prepare the starting material, 729 g of an epoxy-functional methacryl copolymer with an epoxy-weight equivalent of 380 and an average molecular weight (Mn) of 2000, 210 g 1,12-dodecanediacid, 30 g of a light protecting agent (1:1-mixture of Tinuvin® 900 (UV-absorber) and Tinuvin® 111 (sterically hindered amine light protecting agent), both from Ciba Specialty Chemicals, Dettingen, Germany), 3 g benzoin, 6 g Irganox® B 225 (antioxidant, Ciba Specialty Chemicals GmbH, Lampertheim, Germany) and 8 g Troy EX 570 (leveling agent, Troy Chemie GmbH, Seelze, Germany) were first mixed in a dry state and then dispersed at 100 to 120°C by means of an extruder. After cooling off and pre-comminuting of the extrudate, the latter was milled in an air separation ball mill to an average particle size (d50) of 20 µm. The surfactant used was sodium dodecylbenzene sulfonate (SDBS, Pilot Chemical Company, Avenel, New Jersey). In this example, two shear devices (32) were used to produce the desired material. The results appear in Table 1 below.

Example F was prepared in a similar manner to Example D. To prepare the starting material, 500 parts COOH-functional polyester of acid equivalent weight equal to 700 g (available from DSM Resins US, Inc., Augusta, GA) was added to 500 parts of  
 5 bisphenol A epoxy resin of epoxy equivalent weight equal to 700 g (available from Dow Chemical Co., Midland, MI) and 10 parts benzoin (a degassing aid). The surfactant was DBS. The results appear in Table 1 below.

Example G was prepared in a similar manner to Example F, but 300 parts BaSO<sub>4</sub> (available from Polar Minerals, Lawrenceville, GA) were added to the mix. The  
 10 surfactant was SDBS. The results appear in Table 1 below.

11  
 12  
 13  
 14  
 15  
 16  
 17  
 18  
 19  
 20  
 21  
 22  
 23  
 24  
 25  
 26  
 27  
 28  
 29  
 30  
 31  
 32  
 33  
 34  
 35  
 36  
 37  
 38  
 39  
 40  
 41  
 42  
 43  
 44  
 45  
 46  
 47  
 48  
 49  
 50  
 51  
 52  
 53  
 54  
 55  
 56  
 57  
 58  
 59  
 60  
 61  
 62  
 63  
 64  
 65  
 66  
 67  
 68  
 69  
 70  
 71  
 72  
 73  
 74  
 75  
 76  
 77  
 78  
 79  
 80  
 81  
 82  
 83  
 84  
 85  
 86  
 87  
 88  
 89  
 90  
 91  
 92  
 93  
 94  
 95  
 96  
 97  
 98  
 99  
 100  
 101  
 102  
 103  
 104  
 105  
 106  
 107  
 108  
 109  
 110  
 111  
 112  
 113  
 114  
 115  
 116  
 117  
 118  
 119  
 120  
 121  
 122  
 123  
 124  
 125  
 126  
 127  
 128  
 129  
 130  
 131  
 132  
 133  
 134  
 135  
 136  
 137  
 138  
 139  
 140  
 141  
 142  
 143  
 144  
 145  
 146  
 147  
 148  
 149  
 150  
 151  
 152  
 153  
 154  
 155  
 156  
 157  
 158  
 159  
 160  
 161  
 162  
 163  
 164  
 165  
 166  
 167  
 168  
 169  
 170  
 171  
 172  
 173  
 174  
 175  
 176  
 177  
 178  
 179  
 180  
 181  
 182  
 183  
 184  
 185  
 186  
 187  
 188  
 189  
 190  
 191  
 192  
 193  
 194  
 195  
 196  
 197  
 198  
 199  
 200  
 201  
 202  
 203  
 204  
 205  
 206  
 207  
 208  
 209  
 210  
 211  
 212  
 213  
 214  
 215  
 216  
 217  
 218  
 219  
 220  
 221  
 222  
 223  
 224  
 225  
 226  
 227  
 228  
 229  
 230  
 231  
 232  
 233  
 234  
 235  
 236  
 237  
 238  
 239  
 240  
 241  
 242  
 243  
 244  
 245  
 246  
 247  
 248  
 249  
 250  
 251  
 252  
 253  
 254  
 255  
 256  
 257  
 258  
 259  
 260  
 261  
 262  
 263  
 264  
 265  
 266  
 267  
 268  
 269  
 270  
 271  
 272  
 273  
 274  
 275  
 276  
 277  
 278  
 279  
 280  
 281  
 282  
 283  
 284  
 285  
 286  
 287  
 288  
 289  
 290  
 291  
 292  
 293  
 294  
 295  
 296  
 297  
 298  
 299  
 300  
 301  
 302  
 303  
 304  
 305  
 306  
 307  
 308  
 309  
 310  
 311  
 312  
 313  
 314  
 315  
 316  
 317  
 318  
 319  
 320  
 321  
 322  
 323  
 324  
 325  
 326  
 327  
 328  
 329  
 330  
 331  
 332  
 333  
 334  
 335  
 336  
 337  
 338  
 339  
 340  
 341  
 342  
 343  
 344  
 345  
 346  
 347  
 348  
 349  
 350  
 351  
 352  
 353  
 354  
 355  
 356  
 357  
 358  
 359  
 360  
 361  
 362  
 363  
 364  
 365  
 366  
 367  
 368  
 369  
 370  
 371  
 372  
 373  
 374  
 375  
 376  
 377  
 378  
 379  
 380  
 381  
 382  
 383  
 384  
 385  
 386  
 387  
 388  
 389  
 390  
 391  
 392  
 393  
 394  
 395  
 396  
 397  
 398  
 399  
 400  
 401  
 402  
 403  
 404  
 405  
 406  
 407  
 408  
 409  
 410  
 411  
 412  
 413  
 414  
 415  
 416  
 417  
 418  
 419  
 420  
 421  
 422  
 423  
 424  
 425  
 426  
 427  
 428  
 429  
 430  
 431  
 432  
 433  
 434  
 435  
 436  
 437  
 438  
 439  
 440  
 441  
 442  
 443  
 444  
 445  
 446  
 447  
 448  
 449  
 450  
 451  
 452  
 453  
 454  
 455  
 456  
 457  
 458  
 459  
 460  
 461  
 462  
 463  
 464  
 465  
 466  
 467  
 468  
 469  
 470  
 471  
 472  
 473  
 474  
 475  
 476  
 477  
 478  
 479  
 480  
 481  
 482  
 483  
 484  
 485  
 486  
 487  
 488  
 489  
 490  
 491  
 492  
 493  
 494  
 495  
 496  
 497  
 498  
 499  
 500  
 501  
 502  
 503  
 504  
 505  
 506  
 507  
 508  
 509  
 510  
 511  
 512  
 513  
 514  
 515  
 516  
 517  
 518  
 519  
 520  
 521  
 522  
 523  
 524  
 525  
 526  
 527  
 528  
 529  
 530  
 531  
 532  
 533  
 534  
 535  
 536  
 537  
 538  
 539  
 540  
 541  
 542  
 543  
 544  
 545  
 546  
 547  
 548  
 549  
 550  
 551  
 552  
 553  
 554  
 555  
 556  
 557  
 558  
 559  
 560  
 561  
 562  
 563  
 564  
 565  
 566  
 567  
 568  
 569  
 570  
 571  
 572  
 573  
 574  
 575  
 576  
 577  
 578  
 579  
 580  
 581  
 582  
 583  
 584  
 585  
 586  
 587  
 588  
 589  
 590  
 591  
 592  
 593  
 594  
 595  
 596  
 597  
 598  
 599  
 600  
 601  
 602  
 603  
 604  
 605  
 606  
 607  
 608  
 609  
 610  
 611  
 612  
 613  
 614  
 615  
 616  
 617  
 618  
 619  
 620  
 621  
 622  
 623  
 624  
 625  
 626  
 627  
 628  
 629  
 630  
 631  
 632  
 633  
 634  
 635  
 636  
 637  
 638  
 639  
 640  
 641  
 642  
 643  
 644  
 645  
 646  
 647  
 648  
 649  
 650  
 651  
 652  
 653  
 654  
 655  
 656  
 657  
 658  
 659  
 660  
 661  
 662  
 663  
 664  
 665  
 666  
 667  
 668  
 669  
 670  
 671  
 672  
 673  
 674  
 675  
 676  
 677  
 678  
 679  
 680  
 681  
 682  
 683  
 684  
 685  
 686  
 687  
 688  
 689  
 690  
 691  
 692  
 693  
 694  
 695  
 696  
 697  
 698  
 699  
 700  
 701  
 702  
 703  
 704  
 705  
 706  
 707  
 708  
 709  
 710  
 711  
 712  
 713  
 714  
 715  
 716  
 717  
 718  
 719  
 720  
 721  
 722  
 723  
 724  
 725  
 726  
 727  
 728  
 729  
 730  
 731  
 732  
 733  
 734  
 735  
 736  
 737  
 738  
 739  
 740  
 741  
 742  
 743  
 744  
 745  
 746  
 747  
 748  
 749  
 750  
 751  
 752  
 753  
 754  
 755  
 756  
 757  
 758  
 759  
 760  
 761  
 762  
 763  
 764  
 765  
 766  
 767  
 768  
 769  
 770  
 771  
 772  
 773  
 774  
 775  
 776  
 777  
 778  
 779  
 780  
 781  
 782  
 783  
 784  
 785  
 786  
 787  
 788  
 789  
 790  
 791  
 792  
 793  
 794  
 795  
 796  
 797  
 798  
 799  
 800  
 801  
 802  
 803  
 804  
 805  
 806  
 807  
 808  
 809  
 810  
 811  
 812  
 813  
 814  
 815  
 816  
 817  
 818  
 819  
 820  
 821  
 822  
 823  
 824  
 825  
 826  
 827  
 828  
 829  
 830  
 831  
 832  
 833  
 834  
 835  
 836  
 837  
 838  
 839  
 840  
 841  
 842  
 843  
 844  
 845  
 846  
 847  
 848  
 849  
 850  
 851  
 852  
 853  
 854  
 855  
 856  
 857  
 858  
 859  
 860  
 861  
 862  
 863  
 864  
 865  
 866  
 867  
 868  
 869  
 870  
 871  
 872  
 873  
 874  
 875  
 876  
 877  
 878  
 879  
 880  
 881  
 882  
 883  
 884  
 885  
 886  
 887  
 888  
 889  
 890  
 891  
 892  
 893  
 894  
 895  
 896  
 897  
 898  
 899  
 900  
 901  
 902  
 903  
 904  
 905  
 906  
 907  
 908  
 909  
 910  
 911  
 912  
 913  
 914  
 915  
 916  
 917  
 918  
 919  
 920  
 921  
 922  
 923  
 924  
 925  
 926  
 927  
 928  
 929  
 930  
 931  
 932  
 933  
 934  
 935  
 936  
 937  
 938  
 939  
 940  
 941  
 942  
 943  
 944  
 945  
 946  
 947  
 948  
 949  
 950  
 951  
 952  
 953  
 954  
 955  
 956  
 957  
 958  
 959  
 960  
 961  
 962  
 963  
 964  
 965  
 966  
 967  
 968  
 969  
 970  
 971  
 972  
 973  
 974  
 975  
 976  
 977  
 978  
 979  
 980  
 981  
 982  
 983  
 984  
 985  
 986  
 987  
 988  
 989  
 990  
 991  
 992  
 993  
 994  
 995  
 996  
 997  
 998  
 999  
 1000  
 1001  
 1002  
 1003  
 1004  
 1005  
 1006  
 1007  
 1008  
 1009  
 1010  
 1011  
 1012  
 1013  
 1014  
 1015  
 1016  
 1017  
 1018  
 1019  
 1020  
 1021  
 1022  
 1023  
 1024  
 1025  
 1026  
 1027  
 1028  
 1029  
 1030  
 1031  
 1032  
 1033  
 1034  
 1035  
 1036  
 1037  
 1038  
 1039  
 1040  
 1041  
 1042  
 1043  
 1044  
 1045  
 1046  
 1047  
 1048  
 1049  
 1050  
 1051  
 1052  
 1053  
 1054  
 1055  
 1056  
 1057  
 1058  
 1059  
 1060  
 1061  
 1062  
 1063  
 1064  
 1065  
 1066  
 1067  
 1068  
 1069  
 1070  
 1071  
 1072  
 1073  
 1074  
 1075  
 1076  
 1077  
 1078  
 1079  
 1080  
 1081  
 1082  
 1083  
 1084  
 1085  
 1086  
 1087  
 1088  
 1089  
 1090  
 1091  
 1092  
 1093  
 1094  
 1095  
 1096  
 1097  
 1098  
 1099  
 1100  
 1101  
 1102  
 1103  
 1104  
 1105  
 1106  
 1107  
 1108  
 1109  
 1110  
 1111  
 1112  
 1113  
 1114  
 1115  
 1116  
 1117  
 1118  
 1119  
 1120  
 1121  
 1122  
 1123  
 1124  
 1125  
 1126  
 1127  
 1128  
 1129  
 1130  
 1131  
 1132  
 1133  
 1134  
 1135  
 1136  
 1137  
 1138  
 1139  
 1140  
 1141  
 1142  
 1143  
 1144  
 1145  
 1146  
 1147  
 1148  
 1149  
 1150  
 1151  
 1152  
 1153  
 1154  
 1155  
 1156  
 1157  
 1158  
 1159  
 1160  
 1161  
 1162  
 1163  
 1164  
 1165  
 1166  
 1167  
 1168  
 1169  
 1170  
 1171  
 1172  
 1173  
 1174  
 1175  
 1176  
 1177  
 1178  
 1179  
 1180  
 1181  
 1182  
 1183  
 1184  
 1185  
 1186  
 1187  
 1188  
 1189  
 1190  
 1191  
 1192  
 1193  
 1194  
 1195  
 1196  
 1197  
 1198  
 1199  
 1200  
 1201  
 1202  
 1203  
 1204  
 1205  
 1206  
 1207  
 1208  
 1209  
 1210  
 1211  
 1212  
 1213  
 1214  
 1215  
 1216  
 1217  
 1218  
 1219  
 1220  
 1221  
 1222  
 1223  
 1224  
 1225  
 1226  
 1227  
 1228  
 1229  
 1230  
 1231  
 1232  
 1233  
 1234  
 1235  
 1236  
 1237  
 1238  
 1239  
 1240  
 1241  
 1242  
 1243  
 1244  
 1245  
 1246  
 1247  
 1248  
 1249  
 1250  
 1251  
 1252  
 1253  
 1254  
 1255  
 1256  
 1257  
 1258  
 1259  
 1260  
 1261  
 1262  
 1263  
 1264  
 1265  
 1266  
 1267  
 1268  
 1269  
 1270  
 1271  
 1272  
 1273  
 1274  
 1275  
 1276  
 1277  
 1278  
 1279  
 1280  
 1281  
 1282  
 1283  
 1284  
 1285  
 1286  
 1287  
 1288  
 1289  
 1290  
 1291  
 1292  
 1293  
 1294  
 1295  
 1296  
 1297  
 1298  
 1299  
 1300  
 1301  
 1302  
 1303  
 1304  
 1305  
 1306  
 1307  
 1308  
 1309  
 1310  
 1311  
 1312  
 1313  
 1314  
 1315  
 1316  
 1317  
 1318  
 1319  
 1320  
 1321  
 1322  
 1323  
 1324  
 1325  
 1326  
 1327  
 1328  
 1329  
 1330  
 1331  
 1332  
 1333  
 1334  
 1335  
 1336  
 1337  
 1338  
 1339  
 1340  
 1341  
 1342  
 1343  
 1344  
 1345  
 1346  
 1347  
 1348  
 1349  
 1350  
 1351  
 1352  
 1353  
 1354  
 1355  
 1356  
 1357  
 1358  
 1359  
 1360  
 1361  
 1362  
 1363  
 1364  
 1365  
 1366  
 1367  
 1368  
 1369  
 1370  
 1371  
 1372  
 1373  
 1374  
 1375  
 1376  
 1377  
 1378  
 1379  
 1380  
 1381  
 1382  
 1383  
 1384  
 1385  
 1386  
 1387  
 1388  
 1389  
 1390  
 1391  
 1392  
 1393  
 1394  
 1395  
 1396  
 1397  
 1398  
 1399  
 1400  
 1401  
 1402  
 1403  
 1404  
 1405  
 1406  
 1407  
 1408  
 1409  
 1410  
 1411  
 1412  
 1413  
 1414  
 1415  
 1416  
 1417  
 1418  
 1419  
 1420  
 1421  
 1422  
 1423  
 1424  
 1425  
 1426  
 1427  
 1428  
 1429  
 1430  
 1431  
 1432  
 1433  
 1434  
 1435  
 1436  
 1437  
 1438  
 1439  
 1440  
 1441  
 1442  
 1443  
 1444  
 1445  
 1446  
 1447  
 1448  
 1449  
 1450  
 1451  
 1452  
 1453  
 1454  
 1455  
 1456  
 1457  
 1458  
 1459  
 1460  
 1461  
 1462  
 1463  
 1464  
 1465  
 1466  
 1467  
 1468  
 1469  
 1470

FA0984 US NA

TABLE 1

Example	A	B	C	D	E	F	G
Polymer	Nucrel (R) 960	Nucrel (R) RX76	LDPE	Nucrel (R) RX76-Z/GMA	GMA-DDDA	Houston "clear" Polyester-epoxy	Houston "white" Polyester-epoxy
Pigment	-----	-----	-----	-----	-----	-----	BaSO <sub>4</sub>
Pigment loading	-----	-----	-----	-----	-----	-----	30 pph
Polymer type	Thermoplastic	Thermoplastic	Thermoplastic	Thermoset	Thermoset	Thermoset	Thermoset
Melting temperature	95 C	93 C	108 C	-----	-----	-----	-----
Glass transition temperature	-10 C	-10 C	-122 C	97 C	52 C	63 C	63 C
Polymer density	950 kg/m <sup>3</sup>	950 kg/m <sup>3</sup>	920 kg/m <sup>3</sup>	950 kg/m <sup>3</sup>	1450 kg/m <sup>3</sup>	1200 kg/m <sup>3</sup>	1440 kg/m <sup>3</sup>
Polymer loading	0.95 g/cm <sup>3</sup>	0.95 g/cm <sup>3</sup>	0.92 g/cm <sup>3</sup>	0.95 g/cm <sup>3</sup>	1.45 g/cm <sup>3</sup>	1.20 g/cm <sup>3</sup>	1.44 g/cm <sup>3</sup>
Surfactant	10 pph	10 pph	5 pph	5 pph	3 pph	3 pph	3 pph
Surfactant loading	F98	F98	F98	F98	SDBS	SDBS	SDBS
Slurry tank 10	1 pph	0.5 pph	1 pph	1 pph	1 pph	0.5 pph	0.5 pph
Temperature	28 C	28 C	28 C	28 C	28 C	28 C	28 C
Metering pump	89 rad/s	89 rad/s	33 rad/s	44 rad/s	48 rad/s	65 rad/s	78 rad/s
means 22 speed	850 rpm	850 rpm	315 rpm	420 rpm	459 rpm	610 rpm	744 rpm
Metering pump	653 kPa	653 kPa	577 kPa	639 kPa	584 kPa	508 kPa	508 kPa
means 22 pressure	80 psig	80 psig	69 psig	78 psig	70 psig	59 psig	59 psig
Flowrate of mixture at delivery port 24	6.69E-05 m <sup>3</sup> /s	6.69E-05 m <sup>3</sup> /s	4.73E-05 m <sup>3</sup> /s	6.31E-05 m <sup>3</sup> /s	6.94E-05 m <sup>3</sup> /s	9.46E-05 m <sup>3</sup> /s	1.14E-04 m <sup>3</sup> /s
Velocity of mixture at delivery port 24	0.22 m/s	0.22 m/s	0.17 m/s	0.22 m/s	0.24 m/s	0.33 m/s	0.40 m/s
Burand's velocity (V <sub>m</sub> )	0.19 m/s	0.19 m/s	0.17 m/s	0.73 ft/s	0.80 ft/s	1.09 ft/s	1.31 ft/s
Residence time in Heating means 5	9.88 s	9.88 s	12.92 s	9.69 s	8.81 s	6.46 s	5.39 s

Temperature of mixture at output port 30	135 C	100 C	140 C	128 C	130 C	110 C	110 C
Flowrate of mixture at output port 30	9.59E-05 m <sup>3</sup> /s 1.52 gpm	9.59E-05 m <sup>3</sup> /s 1.52 gpm	6.15E-05 m <sup>3</sup> /s 0.98 gpm	8.20E-05 m <sup>3</sup> /s 1.30 gpm	9.02E-05 m <sup>3</sup> /s 1.43 gpm	1.23E-04 m <sup>3</sup> /s 1.95 gpm	1.48E-04 m <sup>3</sup> /s 2.34 gpm
Velocity of mixture at output port 30	0.31 m/s 1.03 ft/s	0.31 m/s 1.03 ft/s	0.22 m/s 0.71 ft/s	0.29 m/s 0.94 ft/s	0.32 m/s 1.04 ft/s	0.43 m/s 1.43 ft/s	0.52 m/s 1.70 ft/s
Shear device 32 speed 1	112 rad/s 1065 rpm	63 rad/s 600 rpm	17 rad/s 159 rpm	52 rad/s 500 rpm	314 rad/s 3000 rpm	22 rad/s 210 rpm	79 rad/s 753 rpm
2	-----	-----	-----	-----	314 rad/s	-----	-----
Shear rate at shear device 32	7645 1/s	4307 1/s	1134 1/s	3589 1/s	21535 1/s	1507 1/s	5405 1/s
Residence time in shear device 32	1.43 s	1.43 s	2.23 s	1.67 s	31790 1/s	1.11 s	0.93 s
Temperature of mixture after cooling means 9	24 C	32 C	16 C	18 C	35 C	37 C	27 C
Flowrate of mixture at exit port 44	9.59E-05 m <sup>3</sup> /s 1.52 gpm	9.59E-05 m <sup>3</sup> /s 1.52 gpm	6.15E-05 m <sup>3</sup> /s 0.98 gpm	8.20E-05 m <sup>3</sup> /s 1.30 gpm	9.02E-05 m <sup>3</sup> /s 1.43 gpm	1.23E-04 m <sup>3</sup> /s 1.95 gpm	1.48E-04 m <sup>3</sup> /s 2.34 gpm
Velocity of mixture at exit port 44	0.31 m/s 1.03 ft/s	0.31 m/s 1.03 ft/s	0.22 m/s 0.71 ft/s	0.29 m/s 0.94 ft/s	0.32 m/s 1.04 ft/s	0.43 m/s 1.43 ft/s	0.52 m/s 1.70 ft/s
Pressure let down pump means 50 speed	63 rad/s 600 rpm	63 rad/s 600 rpm	41 rad/s 390 rpm	55 rad/s 520 rpm	64 rad/s 607 rpm	81 rad/s 775 rpm	95 rad/s 910 rpm
Pressure let down pump means 50 pressure	653 kPa 80 psig	653 kPa 80 psig	529 kPa 62 psig	639 kPa 78 psig	536 kPa 63 psig	508 kPa 59 psig	494 kPa 57 psig
Polymer pellet size	3 mm	3 mm	4 mm	3 mm	20 um average	< 2 mm > 0.125 mm	< 1 mm > 0.125 mm
Polymer particle size	< 100 um	< 50 um	< 10 um > 1 um	< 50 um > 10 um	20 um average	< 100 um > 1 um	< 70 um > 8 um